RIGA TECHNICAL UNIVERSITY
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RESEARCH OF REFERENCE WEIGHT INTERLABORATORY COMPARISONS

Field: mechanical engineering. Subfield: measurement instrumentation and metrology.

Summary of Doctoral Thesis

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DOCTORAL THESIS HAS BEEN SUBMITTED TO RIGA TECHNICAL UNIVERSITY FOR OBTAINING OF ENGINEERING SCIENCES DOCTOR SCIENTIFIC DEGREE

Doctoral thesis for obtaining engineering sciences doctor scientific degree will be defended on March 02, 2011 at 15:00 in the Riga Technical University, faculty of Transport and Engineering Science, address: Ezermalas iela 6, room 405.

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CONFIRMATION

I confirm that I have created this thesis submitted to Riga Technical University, to obtain doctor of engineering degree. This doctoral thesis has not submitted to other university for obtaining of scientific degree.

Tatjana Ivanova …….(Signature)

Date: ……

The thesis is written in Latvian language and consists of introduction, four chapters, resume, references and appendixes. It is written in 117 pages and contains 29 figures, 69 tables, 3 appendixes and 25 references.

TABLE OF CONTENTS

participants have problems with calculation of measurement uncertainty and best measurement capability. Laboratories assess their own abilities too optimistically and do not observe requirements on international recommendation.

4. During calibration process in the pilot laboratory, we have discovered that mass comparator is highly susceptible to power supply disturbances. Changes in voltage during calibration have influenced ABBA circle results and therefore measurements failed. When voltage was relatively stable measurements were successful (“ok” in calibrating protocol). Quality of electrical power supply could be checked using regular digital multimeter in alternating current measurement mode. This problem was discovered during experiment and was not described in “Sartorius” documents. This problem may be eliminated by improvement of power supply. For example, double conversion UPS (uninterruptible power supply) can be installed. It could reduce time needed for every measurement and therefore increase productivity of the laboratory.

5. Given method of mutual comparison gives new opportunities to discover not only errors of leading laboratories but also see errors of laboratories that can not participate in mutual comparison of world laboratories. This method allows discovery of errors of participating laboratories that could remain unnoticed and therefore disturb both economy of the country and medicine and pharmacy industries. This paper gives opportunity to improve mass measurement system in Latvia.

standard of mass) – it is possible that participating laboratories had problems with power supply as it can significantly influence stability of measurements.

3. Component C is also mentioned in measurement function that observes influence of aerostatic forces on measurement process. It is possible that air density and density of the weight material do not correspond to requirements of international recommendation OIML R111.

4. Fourth component Δm_d - error of measurement equipment. Error of measurement equipment is influenced by magnetic forces, eccentricity and other noises. For example, during measurement process it is forbidden to use mobile phones that can influence measurement process.

CONCLUSIONS

Following conclusions can be done:

1. For the first time in Latvia interlaboratory comparison was done in the field of mass measurement. Interlaboratory comparisons were done between five accredited laboratories of Latvia including pilot laboratory (State agency of metrology and accreditation).

2. Interlaboratory comparison method was developed and this method differs from existing ones. This method gives opportunity to conduct interlaboratory measurements not only in the field of mass measurements but also in other fields of metrology. This method best suits to laboratories that are not leading in the world. Using this method, laboratories have possibility to evaluate not only participating laboratories but also their own abilities.

3. Following interlaboratory measurement results were received:

a) Laboratory „A" calculated very low measurement uncertainty for nominal mass values of 50 g and 1 kg, that generated unacceptable value of E_n. There are technical problems with nominal of 20 kg, as increase of uncertainty up to permissible limit (1/3 from permissible error) can not decrease E_n to acceptable level. The best measurement capability for nominal values of 500 g, 1 kg and 20 kg was not determined correctly.
b) Laboratory „B" has best measurement capability for nominal of 20 kg that does not correspond to accuracy class from accreditation certificate.
c) Laboratory „C" results in general do not correspond to the accuracy class from accreditation certificate.
d) Laboratory „D" incorrectly determined best measurement capability for all nominal values.

Results of participating laboratories generally can be regarded as satisfactory from technical point of view (E_n coefficient). However, almost all

THESIS OUTLINE

Significance of research

Topic of this thesis corresponds to one of the main objectives of State Agency of metrology and accreditation of Latvia (MAVA): project of interlaboratory comparison. Participation in this project is necessary according to law of Latvia "On the Assurance of Measurement Uniformity". Participation in such project is required for accredited measurement laboratories in order to validate competency and ensure quality by European standard EN ISO/IEC 17025:2005 „General requirements for the competency of testing and calibration laboratories".

The experience acquired in such interlaboratory comparisons can be useful also in other engineering metrology studies.

Goals and objectives of the paper

The goal of this paper is to develop a method that would help controlling institutions to inspect the ability of accredited laboratories of mass measurement to conduct precise measurements. This can be done by comparing results of pilot laboratory (MAVA) with results of participating laboratories.

The topic of this thesis is related to the organization of interlaboratory comparison in the area of mass measurement. This has not been done in Latvia yet.

Therefore following objectives have to be solved in this promotion paper:

1) Analysis of equipment for high precision mass measurement and analysis of weighting methods;
2) Development of mathematical model for mass measurement and determination of measurement uncertainty;
3) Development of scheme for interlaboratory comparison;
4) Development of method that determines reference value and measurement uncertainty;
5) Selection of data analysis method;
6) Practical application of developed method of interlaboratory comparison.

Research method

Following research methods were used in this paper:
1) Determination of measurement uncertainty according to JCGM 104:2009 guidelines;
2) „Scales Net 32" software was used for initial processing of laboratory results;
3) Statistical methods, probability theory and errors analysis.

Scientific novelty and main results of the research

For the first time in Latvia an interlaboratory comparison among accredited laboratories of mass measurement was conducted. Laboratories that provide services of weights and scales calibration and verification participated in comparison.

A method with star-shaped scheme for interlaboratory comparison was developed in this paper. This method can also be used for interlaboratory comparisons in other fields. The advantage of this method is the ability to detect changes in mass of the standard of mass between participating laboratories. A mathematical model to determine mass measurement uncertainty was developed.

Comparison operations are very important for traceability of the value of mass measurement and to comply with international standards. Both precision and validity of laboratory results were estimated during research. It was discovered that some participants of the interlaboratory comparison should improve accuracy and validity of their mass measurements.

Practical application

The result of the research may be used to improve the quality of mass measurements in laboratories of Latvia. That could enhance competitiveness of laboratories and affirm Latvia positions in European metrology. Method for estimating reference value and comparison of different laboratories' results can be used for conducting interlaboratory comparisons in other countries.

With little changes, this method can be used for interlaboratory comparisons also in other areas of physical value measurements.

Table 3

Comparison of actual uncertainty and best measurement capability (BMC)

Laboratories	**Laboratory "A" accr. for class M_1**		**Laboratory "B" accr. for class F_1**		**Laboratory "C" accr. for class F_1**		**Laboratory "D" accr. for class F_1**	
	Calibration results		Calibration results		Calibration results		Calibration results	
Nominal mass	Conventional mass	**Act. uncertainty and BMC ratio, *U (%)***	Conventional mass	**Act. uncertainty and BMC ratio, *U (%)***	Conventional mass	**Act. uncertainty and BMC ratio, *U (%)***	Conventional mass	**Act. uncertainty and BMC ratio, *U (%)***
100 mg	0,012 mg	**18,8**	-0,001 mg	**10,6**	0,013 mg	**not accr.**	0,0097 mg	**not accr.**
1 g	0,007 mg	**14,8**	0,001 mg	**12,1**	-0,03 mg	**111,4**	0,002 mg	**104,0**
50 g	0,033 mg	**16,8**	-0,060 mg	**21,0**	-0,16 mg	**111,0**	-0,073 mg	**113,8**
100 g	0,011 mg	**23,2**	0,034 mg	**10,0**	0,01 mg	**118,5**	0,055 mg	**127,8**
500 g	1,090 mg	**121,8**	0,303 mg	**9,8**	-0,2 mg	**109,3**	0,449 mg	**1090,9**
1 kg	3,184 mg	**123,7**	-0,385 mg	**10,0**	-1,0 mg	**108,2**	-1,0 mg	**not accr.**
20 kg	339,73 mg	**130,1**	-16 mg	**14,9**	9,51 mg	**142,3**	49 mg	**not accr.**

From table 3 it is possible to conclude that laboratory "A" for nominal mass values 500 g, 1 kg, 20 kg; laboratories "C" and "D" for almost all nominal mass values have big uncertainty regarding best weighting method. Laboratories have to pay attention to determine the best weighting method.

Table 4 displays problematic results of participating laboratories.

Table 4

Results analysis
("+"-satisfactory result, "–"-non-satisfactory result)

Nominal mass	**Laboratory „A"**		**Laboratory „B"**		**Laboratory „C"**		**Laboratory „D"**	
	E_n	Uncer-tainty	E_n	Uncer-tainty	E_n	Uncer-tainty	E_n	Uncer-tainty
100 mg	(+)	(+)	(+)	(+)	(+)	**(–)***	(+)	**(–)***
1 g	(+)	(+)	(+)	(+)	(+)	**(–)**	(+)	**(–)**
50 g	**(–)**	(+)	(+)	(+)	(+)	**(–)**	(+)	**(–)**
100 g	(+)	(+)	(+)	(+)	(+)	**(–)**	(+)	**(–)**
500 g	(+)	**(–)**	(+)	(+)	(+)	**(–)**	(+)	**(–)**
1 kg	**(–)**	**(–)**	(+)	(+)	(+)	**(–)**	(+)	**(–)***
20 kg	**(–)**	**(–)**	(+)	**(–)**	(+)	**(–)**	(+)	**(–)***

After conducting data analysis possible reasons for differences in participating laboratories results are described in chapter 4. Differences in laboratories' measurements can be assessed using measurement function of conventional mass (Fig.5), where $m_{ct} = m_{cr} + \Delta I + C + \Delta m_d$. All of these components can influence measurement results. If it is known that m_{cr} - is conditional mass of the standard of mass.

1. Every participating laboratory used its own standards of mass for interlaboratory measurements. It is possible that standards of mass had big uncertainty. We can not exclude that external environment conditions of participating laboratories (temperature, relative humidity of air and others) did not correspond to requirements of international recommendation.

2. Regarding second component ΔI (differences between test weight and

4.7. Analysis of the expanded uncertainty and best measurement capability in participating laboratories

Now it is necessary to evaluate expanded uncertainty in every laboratory. In order to evaluate uncertainty it is necessary to apply recommendation OIML R111 which allows to calculate allowed uncertainty for every weight depending on accuracy class. Allowed uncertainty is equal to one third of allowed error: $U_{perm.} = \frac{1}{3}\sigma$. Uncertainty can be calculated in percents for every laboratory and compared to allowed uncertainty (Table 2). If we assume that allowed uncertainty is 100% then we can calculate how many percents of uncertainty was obtained in every laboratory. For instance laboratory "A" determined uncertainty was 0.032 mg for 100 mg weight, but allowed uncertainty is 0.166 mg (1/3 from negligible error 0.5 mg). If allowed uncertainty 0.166 mg is 100% than relative uncertainty is 100*0.032/0.166=19.2%. Relative uncertainty can be calculated for other laboratories in the same way.

Table 2

Comparison of actual and permissible uncertainty according to international recommendation OIML R111

Laboratories	**Laboratory "A" accr. for class M_1**		**Laboratory "B" accr. for class F_1**		**Laboratory "C" accr. for class F_1**		**Laboratory "D" accr. for class F_1**	
	Calibration results		Calibration results		Calibration results		Calibration results	
Nominal mass	Conventional mass	**Act. and perm. uncertainty ratio *U (%)***	Conventional mass	**Act. and perm. uncertainty ratio *U (%)***	Conventional mass	**Act. and perm. uncertainty ratio *U (%)***	Conventional mass	**Act. and perm. uncertainty ratio *U (%)***
100 mg	0,012 mg	**19,2**	-0,001 mg	**10,2**	0,013 mg	**852,0**	0,0097 mg	**156,0**
1 g	0,007 mg	**9,3**	0,001 mg	**12,0**	-0,03 mg	**486,0**	0,002 mg	**78,0**
50 g	0,033 mg	**4,2**	-0,060 mg	**21,0**	-0,16 mg	**222,0**	-0,073 mg	**33,0**
100 g	0,011 mg	**3,5**	0,034 mg	**9,6**	0,01 mg	**184,8**	0,055 mg	**27,6**
500 g	1,090 mg	**19,0**	0,303 mg	**9,7**	-0,2 mg	**550,9**	0,449 mg	**14,4**
1 kg	3,184 mg	**12,6**	-0,385 mg	**9,6**	-1,0 mg	**298,6**	-1,0 mg	**13800,0**
20 kg	339,73 mg	**35,5**	-16 mg	**44,7**	9,51 mg	**175,5**	49 mg	**690,0**

It is possible to conclude from table 2 that laboratories "C" and "D" have large uncertainty that is not allowed.

Next, it is necessary to assess best measurement capabilities of participating laboratories that can be found in accreditation documents. Then it is possible to calculate in percents how big the uncertainty in every laboratory is by comparing with best measurement capabilities of participating laboratories (table 3).

In this paper author defends:

1) Developed method of interlaboratory mass measurement;
2) Practical application of the method:
   a) reference value and measurement uncertainty determination for weights of nominal mass: 100 mg, 1g, 50 g, 100 g, 500g, 1 kg, 20 kg, using mass comparator;
   b) evaluation of measurements results.

Paper approbation

Notifications on main success points, results of the thesis and positive appraisals were received from following conferences and seminars:

<u>In Latvia:</u>

- 49th International Science conference of Riga Technical University 13.10. – 15.10.2008.
- 50th International Science conference of Riga Technical University 12.10. – 16.10.2008.
- RTU TMF P-16 seminar of promotion board. Chief of the science seminar – professor Jānis Rudzītis. Riga. 15 October, 2009.
- RTU TMF scientific seminar of Institute of mechanics. Chief of the science seminar – professor Jānis Vība. Riga. 15 December, 2009.

<u>In foreign countries:</u>

- 4 th International Conference Mechatronic Systems and Materials 14.07-17.07.2008, Bialystok, Poland.
- 5 th International Conference Mechatronic Systems and Materials 22.10-25.10.2009, Vilnius, Lithuania.
- International Congress – Machines, Technologies, Materials MTM 2010 26.05-28.05.2010, Sofia, Bulgaria.
- 7th International Conference on Bionics and Prosthetics, Biomechanics and Mechanics, Mechatronics and Robotics VARNA'2010, 24.05-25.05.2010, Liepaya, Latvia.
- 6th International Conference Mechatronic Systems and Materials 05.07.-08.07.2010, Opole, Poland.

Publications

There were six scientific articles published about research results and developments:

1. Ivanova T., Rudzitis J. Traceability and Capability Control of Mass Measurement Equipment and Drift Statistical Analysis of National Mass Standards in Latvia// ISSN 1898-4088. Acta mechanica et automatica. Volume 2. – no.3. - Bialystok: Bialystok Technical University, 2008. - pp. 42-50.
2. Ivanova T., Rudzitis J. High Precision Mass Measurement in Automation// ISSN 1012-0394. Solid State Phenomena. Volume 164. - Switzerland: Trans Tech Publications, 2010. - pp. 19-24.
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4. Иванова Т. Межлабораторное сравнение лабораторий измерения массы в Латвии// ISSN 1313-0226. Международный виртуальный журнал Машины, Технологии, Материалы. № 4-5. - София: Научно-технический союз машиностроения, 2010. - стр. 60-62.
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Structure and volume of the paper

The thesis is written in Latvian language and consists of introduction, four chapters, resume, references and appendixes. It is written in 117 pages and contains 29 figures, 69 tables, 3 appendixes and 25 references.

4.6.2. Analytical evaluation of participating laboratories measurement results

To validate conclusions that are received from graphics, also normalized deviation E_n is used of measurement comparison that is defined as:

$$E_n = \frac{(m_i - m'_{PL})}{\sqrt{U^2(m_i) + U^2(m'_{PL})}} \qquad (12)$$

where $E_{n,i}$ - normalized deviation of *i*-th laboratory.

$U(m_i)$ - expanded uncertainty that is assigned to the *i*-th laboratory and its result m_i,

m'_{PL} - conventional mass in the pilot laboratory,

$U(m'_{PL})$ - expanded uncertainty of conventional mass that is obtained in the pilot laboratory.

For successful accreditation laboratory needs $|E_{n,i}| \le 1$. Therefore we can calculate normalized deviation E_n for every laboratory:

- If $|E_{n,i}| \le 1$, result is satisfactory.
- If $|E_{n,i}| > 1$, result is not satisfactory.

As an analytical example we can view chess-type measurement results of the participating laboratory for the standard of mass of 1 kg (table 1). Here we can see that laboratory "A" result significantly differs from pilot laboratory results.

Table 1

Calculation of E_n numbers (1 kg

LAB_i	PL	A	B	C	D
PL	-	**1,64**	-0,22	-0,14	0,00
A	**-1,64**	-	-1,69	-0,77	-0,02
B	0,22	1,69	-	-0,12	0,00
C	0,14	0,77	0,12	-	0,00
D	0,00	0,02	0,00	0,00	-

4.6. Data evaluation by comparing results of pilot laboratory with participating laboratories

4.6.1. Graphical evaluation of participating laboratories measurements results

Results from all laboratories including pilot laboratory "PL" can be gathered and displayed graphically or in the table. On the *x* axis laboratories including pilot laboratory are shown. Point on the graph (*y* axis) denotes obtained value of the conventional mass (Δm) in all laboratories including pilot laboratory. Vertical line (*y* axis) denotes obtained value of the expanded uncertainty of the conventional mass $U(m)$.

As an example we can see graph of the measurement results of standard of mass of 1 kg in the participating laboratories (Fig. 8). On Figure 8 we can see that results of laboratory "A" tip out from pilot laboratory results, laboratories "C" and "D" have too big measurement uncertainty that is not allowed.

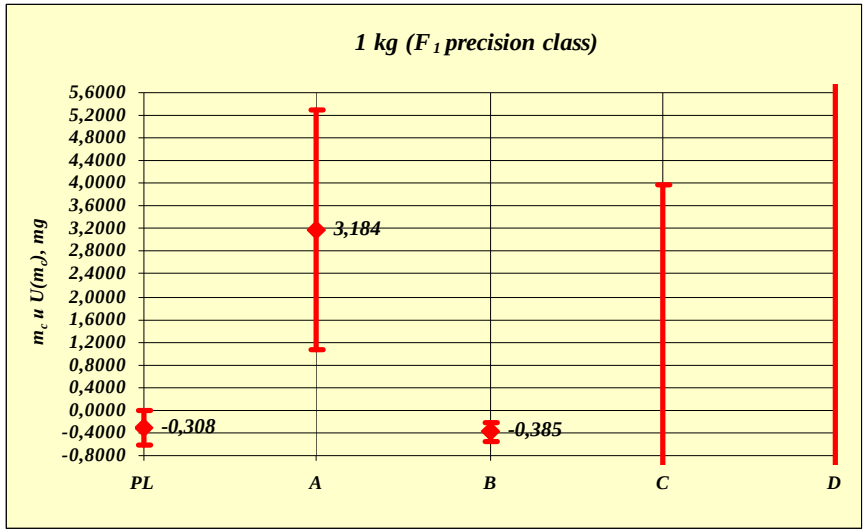


Fig. 8. Calibration results of the standard of mass of 1 kg in participating laboratories

THESIS SUMMARY

Main terms

Following terms are used in this paper:

1) ***Calibration*** – set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.
2) ***Conventional mass*** – conventional value of the result of weighting in air, in accordance with OIML D 28 "Conventional value of the result of weighing in air". For a weight taken at a reference temperature (t_{ref}) of 20 ^{0}C, the conventional mass is the mass of a reference weight of a density (ρ_{ref}) = 8000 kg/m^3 which it balances in air of a reference density (ρ_0) = 1.2 kg/m^3.
3) ***Measurement uncertainty*** – component of measurement results that indicates value range that includes true value.
4) ***Reference value*** – value obtained by the pilot laboratory that is considered to be the closest to the true value; values obtained in participating laboratories are compared to the reference value.
5) ***Pilot laboratory (Reference laboratory)*** – laboratory that conducts interlaboratory measurement comparison and defines reference value of measured unit.
6) ***Participating laboratories*** – participants of interlaboratory comparison.

Introduction

Main concepts of interlaboratory measurement results comparison are reviewed and importance of the topic is affirmed in the introduction. General objectives that have to be solved in interlaboratory measurement comparison process are described. Latvian accredited laboratories of mass measurement and their facilities are reviewed.

Chapter 1 ANALYSIS OF EXSISTING RESULTS OF MASS STANDARD INTERLABORATORY COMPARISONS

Review of previous work in this field has been made. Also, existing standards and recommendations has been reviewed:

1) Latvian law „On measurement uniformity" of 27.02.1997;
2) European standard ISO/IEC 17025:2005 „General competency requirements for testing and calibrating laboratories";
3) ISO/IEC Guide 43 „Proficiency testing by interlaboratory comparisons". This document consists of two parts:

Part 1: Development and operation of proficiency testing schemes;

Part 2: Selection and use of proficiency testing schemes by laboratory accreditation bodies.

Main formal requirements are described in this document including all steps of interlaboratory measurements: development of the scheme, selection of methods, selection of personnel, selection of samples to be measured, transportation of the sample, processing of the results, reports etc. Attention is also paid to topics like confidentiality of results and counterfeiting of measurement results.

Goals and objectives of metrology institutions are also reviewed. One of the main objectives is organization and participation in interlaboratory comparisons.

According to these objectives it is clear that this paper corresponds to main operational directions of metrology institutions.

Foreign interlaboratory comparisons that were conducted in Mexico, Sweden and United Kingdom were reviewed.

Conducted literature analysis shows that previously mentioned countries' researches can not be used in Latvia without corrections. Therefore following objectives are highlighted in this thesis:

1) Analysis of weighting methods and analysis of equipment for high precision mass measurement;
2) Development of mathematical model for mass measurement and measurement uncertainty;
3) Development of interlaboratory comparison scheme;
4) Development of the method for reference value and measurement uncertainty determination;
5) Selection of data analysis method;
6) Practical application of developed interlaboratory comparison method.

where $s'(\Delta m'_{PL}) = \sqrt{\sum_{i=1}^{n} \frac{(\Delta m'_{PLi} - \Delta m'_{PL})^2}{n-1}}$ - standard deviation.

Type "B" component of the standard uncertainty:

- Standard uncertainty of the reference standard of mass, $u(m_{cr})$

It is calculated by dividing expanded uncertainties of corresponding certificate of calibration, *U*, by coverage factor *k* (usually *k*=2). As a result we get standard uncertainty that is combined with uncertainty that is related to instability of reference weight $u_d(m_{cr})$. In order to guarantee accuracy of the results lets take the largest standard uncertainty of the standard of mass *U'* from several reference weights and its largest instability (drift) uncertainty of the reference weight:

$$u'(m_{cr}) = \sqrt{(\frac{U'_{\max}}{k})^2 + u^2{}_d(m_{cr})_{\max}} \quad (8)$$

- Uncertainty of the aerostatic force influence, u'_b is calculated in the following way:

$$u_b = (m_{cr} \cdot \frac{(\rho_r - \rho_t)}{\rho_r \rho_t} \cdot u(\rho_a)) \quad (9)$$

where ρ_r, ρ_t - density of reference and test weights,

ρ_a - air density

- Uncertainty of the scales or mass comparator – can be taken from previous calibration certificate
- Combined standard uncertainty for standard of mass:

$$u_{PL}(\Delta m'_{PL}) = \sqrt{u_w^2(\Delta m'_{PL}) + u^2(m'_{cr}) + u'^2_b + u'^2_{ba}} \quad (10)$$

- Expanded uncertainty of the conventional mass of the standard of mass, *U(m'PL)*,:

$$U(m'_{PL}) = k \cdot u_{PL}(m'_{PL}) \quad (11)$$

Note: general formulas are defined in the International Recommendation OIML R111.

Conventional mass for other standards of mass is calculated in the same way taking into consideration measurement uncertainty.

4.5. Determination of reference value of the standard of mass in the pilot laboratory

4.5.1. Determination of conventional mass of the standard of mass

Let's assume that obtained results of calibration are normally distributed. If we use validity confidence level 99%, filtration can be done according to the following formula:

$$(\Delta m_{PL} - t_{\frac{\alpha}{2}} \cdot \frac{S(\Delta m_{PL})}{\sqrt{n}},\ \Delta m_{PL} + t_{\frac{\alpha}{2}} \cdot \frac{S(\Delta m_{PL})}{\sqrt{n}}) \qquad (4)$$

where $\Delta m_{PL} = \frac{1}{n}\sum_{i=1}^{n} \Delta m_{PLi}$ - average value of the conventional mass,

n - number of calibration results (set size),

$S(\Delta m_{PL})$ - standard deviation of the set,

$t_{\frac{\alpha}{2}}$ - value of Student distribution with *n-1* degrees of freedom that splits off area from Student distribution density function:

$$S(\Delta m_{PLi}) = \sqrt{\sum_{i=1}^{n} \frac{(\Delta m_{PLi} - \Delta m_{PL})^2}{n-1}} \qquad (5)$$

Let's choose conventional mass data that are inside the confidence interval. From these filtered results we can calculate conventional mass as arithmetic mean:

$$\Delta m'_{PL} = \frac{1}{n}\sum_{i=1}^{n} \Delta m'_{PLi} \qquad (6)$$

4.5.2. Determination of the expanded uncertainty of the conventional mass of the standard of mass

If we use measurement uncertainty scheme (mathematical model) from Fig. 5, we can evaluate expanded uncertainty of the conventional mass. Expanded uncertainty is calculated using non-filtered results of calibration.

Type "A" component of the standard uncertainty:

- Standard uncertainty of the weighting process:

$$u_w(\Delta m'_{PL}) = \frac{s'(\Delta m'_{PL})}{\sqrt{n}} \qquad (7)$$

Chapter 2 HIGH PRECISION MASS STANDARDS AND MEASUREMENT EQUIPMENT

Interlaboratory comparisons in the field of mass measurement are conducted using high accuracy mass measurement equipment – weights, scales and mass comparators.

Portable artifacts (standards of mass) are described. In the field of mass measurement, weights of accuracy classes E_1, E_2, F_1, F_2, M_1, $M_{1\text{-}2}$, M_2 and $M_{2\text{-}3}$ are used (Fig. 1).

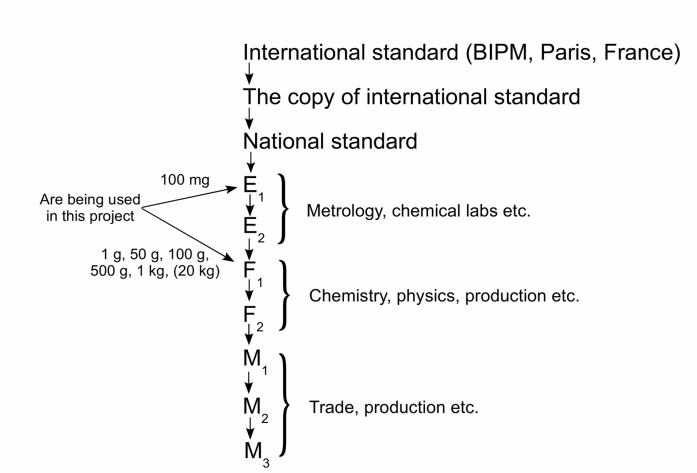


Fig. 1. Weight accuracy classes

E_1 accuracy class weights are meant to ensure traceability among national standards of mass that have their masses obtained from International kilogram prototype (fig. 2) and for E_2 and lower accuracy class weights.

E_2 accuracy class weights are meant for F_1 accuracy class weights verification or calibration and for use with special I accuracy class scales, chemical analysis, accurate weighting in science and engineering.

F_1 accuracy class weights are meant for F_2 accuracy class weights verification and calibration and for use with special I and high II accuracy class scales, chemical analysis, accurate weighting in science and engineering.

F_2 accuracy class weights are meant for M_1 and possibly M_2 accuracy class weights verification or calibration and for use with high II accuracy class scales, technical analysis of advanced accuracy, weighting in science and engineering, weighting of precious metals and gemstones.

M_1 accuracy class weights are meant for M_2 accuracy class weights verification or calibration, for use with medium III accuracy class scales, technical analysis of normal accuracy, weighting in engineering and medicine.

M_2 accuracy class weights are meant for M_3 accuracy class weights verification or calibration, for use in normal commercial operations and with medium III accuracy class scales.

M_3 accuracy class weights are meant for use with normal IIII accuracy class scales.

M_{1-2} and M_{2-3} accuracy class weights are low accuracy weights with nominal mass from 50 kg till 5000 kg that are meant for use with medium III accuracy scales.

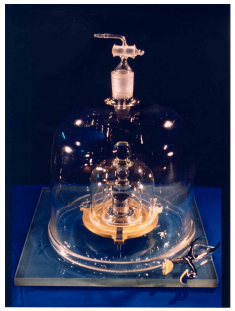


Fig.2. International kilogram prototype

In interlaboratory comparison E_1 accuracy class standards of nominal mass 100 mg and F_1 accuracy class standards of nominal mass 1 g, 50 g, 100 g, 1 kg (20 kg) are used.

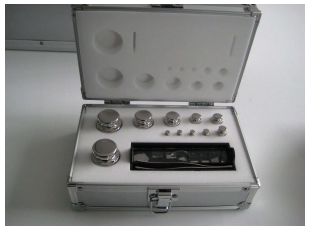


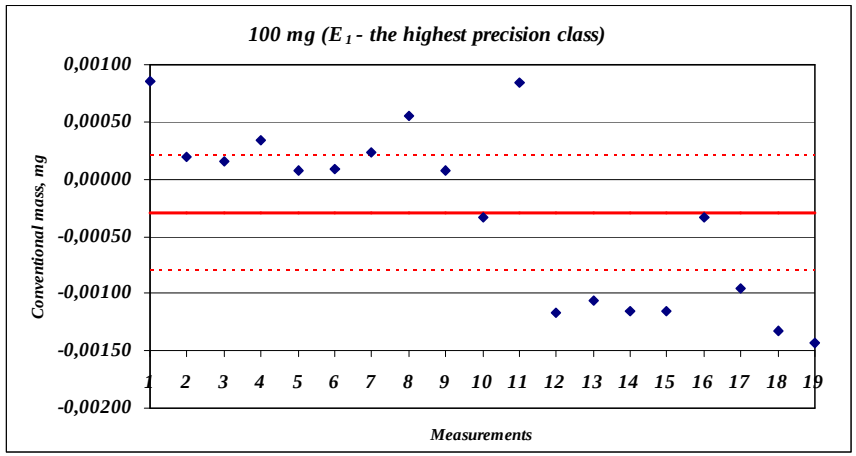
Fig. 3. Typical set of F_1 accuracy class weights

Weights must have no sharp corners and sides in order to avoid possibility of damages, there should not be any significant cavities so that no residue (dust for example) forms on the surface of the weights. Weights that are lighter than 1 g must be manufactured from polygonal plates or wires and their

4.4. Assessing results of mass measurement in pilot laboratory

In order to evaluate stability of the standards of mass before giving them to participating laboratories weights are calibrated *n* times and 2 times after receiving them from participating laboratories. In order to compare results of participating laboratories pilot laboratory has to determine reference values of the standards of mass – conventional mass with measurement uncertainty. Then pilot laboratory sends standards of mass to participating laboratories according to weights turnover scheme.

After last calibration these measurements can be depicted as graph or displayed as a table. As an example we can see results of calibration of the weight 100 mg (Fig. 7)



Confidence level 99%

Fig. 7. Results of conventional mass calibration using filtration

Let's assume that some results are incorrect. The reason of error can be low-quality power supply, air flows, shaking of the foundation of the building where mass comparators are situated but main goal of the pilot laboratory is to determine reference value that would be closest to the true value.

measurement equipment (weights, scales, mass comparators) and their own premises for measurements and applied certified calibrating procedures (methods).

4.2. Rules of interlaboratory measurement comparison.

Pilot laboratory has to observe formal requirements in regard of scheme development, choosing method, personnel selection, choosing research sample, transportation of the sample, processing of results. Attention has also to be paid to confidentiality and results forgery.

4.3. The choice of the interlaboratory measurement comparison scheme depending on number of participating laboratories and their geographical location.

The most regular schemes of weights turnover are ring-type and star-type schemes. As number of participants is small pilot laboratory used star-type turnover scheme (Fig. 6) to inspect mass of the weights after every participating laboratory.

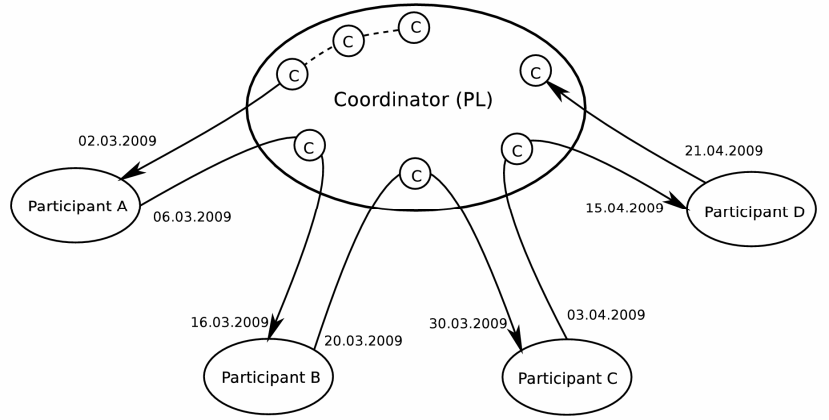


Fig. 6. Standards of mass turnover scheme

Laboratories „A", „B", „C" and „D" participated in interlaboratory measurement comparison. Laboratories agreed to develop schedule of participation. In the end of the turnover the pilot laboratory repeatedly calibrates standards of mass and analyses all results taking into consideration measurement uncertainties reported by participating laboratories.

form should be adjusted to the most convenient use. Particular form denotes nominal mass of the weight. Under normal use conditions surface of the weights should be in such condition that ensures that changes in the mass of weights are insignificant compared to the given class of accuracy and maximal acceptable error. It can be ensured by using appropriate surface protection method. Surface of the weights is examined visually.

Attention is also paid to conditions of weighting. Accuracy of weighting depends not only on weights accuracy, scales and measurement methods but also on environmental conditions.

Weighting premises should be placed not higher than ground floor on the north side of the building and far from busy roads. Premises should be dry and light. Laboratory premises for weights should include two rooms: preparation and weighting rooms. In the preparation room weights are unpacked, their surface is checked then weights are cleaned and prepared for measurement.

Weighting room should have special foundation that is not connected to the floor. Marble plates should cover foundation. Size of the plates can be different depending on the size of the scales that will be placed on them. Plates for scales can be manufactured also from other materials that ensure even and smooth surface and do not deform under the weight of the scales.

There should be no sinks, water pipes, heating or wastewater pipes in the premises. Ventilation channels and air conditioners should be placed so that no convection flows are created. Constant presence of employees in this premise is not advisable therefore number of working places should be minimal. There should be additional equipment for measurements of atmosphere pressure, humidity, temperature. According to OIML R111conditions of external environment during calibration process are highlighted in this paper. The temperature range for E_1 and E_2 accuracy class weights is from 18 ^{0}C till 27 ^{0}C, humidity range is from 40% till 60%.

Nowadays mechanical scales are replaced with electronic ones. The main principle of electronic scales operation is transformation of force of gravity into electrical signal. Parameters of the signal (current, voltage or frequency) are measured with common electrical methods and they are recast into mass units. Measured value can be displayed on the scales screen or it can be sent for further processing to the computer. Moreover, many electronic scales have additional functions for processing of measured values: summing, counting of measurements, price calculation, results comparison etc.

Mass comparators are special high accuracy scales that are meant for mass determination using comparison method. Mass of the test sample is

compared with standard of mass. In order to do it standard of mass (A) and test weight (B) are weighted several times in series. After that, a conditional mass is calculated.

Mass comparators use sensor with electromagnetic compensation that ensures high accuracy of measurements. Construction of the comparator is possibly massive and rigid. Comparators are divided into automatic and manual. Automatic comparators have special mechanism for shifting weights A and B during measurement circles ("carousel"). Manual comparators do not have such mechanism and operator has to switch weights manually during measurement process. Automatic comparators are more accurate than manual ones as automatic are less influenced by inaccuracy of operator activities and his movements in the proximity of comparator. Moreover, automatic comparators have delayed weighting function that allows conduct measurements at the night when vibration of building and fluctuation of voltage are minimal. The most accurate comparators are equipped with vacuum camera where measurements are conducted. Therefore influence of the air is excluded from measurements results.

Comparators are high accuracy scales with a very high resolution (tens millions of discreet units). Such scales can measure mass difference between two objects more accurate than the mass of these objects. Moreover, if the mass of one object is already known it is possible to calculate the mass of second object by adding to known mass the difference of masses. Here is a simplified example. Let's assume that mass of two weights (A and B) measured by comparator is 975 and 977 mg. It is known that accurate mass of the weight A is 1001 mg. Now we can easily calculate accurate mass of the weight B: 1001+(977-975)=1003 mg.

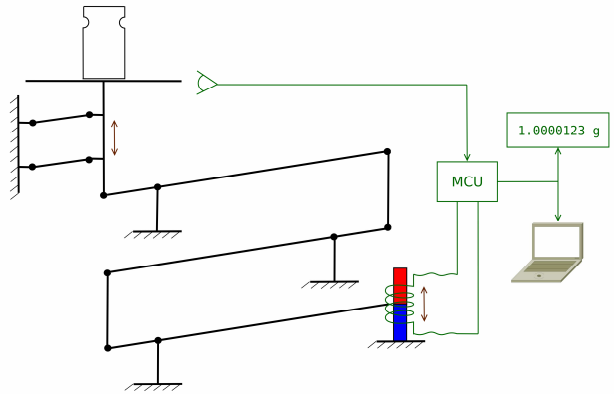


Fig. 4. Functional scheme of mass comparator

Chapter 4 INTERLABORATORY COMPARISON METHOD

Interlaboratory comparison method is described in chapter 4. Based on previous projects on interlaboratory comparison a method with a star-type scheme was developed for mass measurement.

Interlaboratory measurement comparison is assessment of results data between pilot laboratory and participating laboratories. Pilot laboratory (MAVA) for the first time in Latvia organized comparisons between accredited laboratories of Latvia. The aim of interlaboratory measurement comparison is to assess competency of accredited laboratories or laboratories that soon will be accredited in Latvia, assess their facilities in weights calibration in order to ensure quality of the service and validity of the results.

Interlaboratory measurement method development has to include following stages:

1. Choosing measurement object and specifying assignments for participating laboratories.
2. Rules of interlaboratory measurement comparison.
3. The choice of the interlaboratory measurement comparison scheme depending on number of participating laboratories and their geographical location.
4. Assessing results of mass measurement in pilot laboratory.
5. Determination of reference value of the standard of mass in the pilot laboratory:
   a) Determination of conventional mass of the standard of mass.
   b) Determination of the expanded uncertainty of the conventional mass of the standard of mass.
6. Data evaluation by comparing results of pilot laboratory with participating laboratories:
   a) Graphical evaluation of participating laboratories measurements results.
   b) Analytical evaluation of participating laboratories measurement results.
7. Analysis of the expanded uncertainty and best measurement capability in participating laboratories.

4.1. Choosing measurement object and specifying assignments for participating laboratories

A set of standards of mass (weights) is chosen to be the object of measurements. Pilot laboratory gave assignment to participating laboratories to determine conditional mass of the weights taking into consideration measurement uncertainty. Participating laboratories used their own

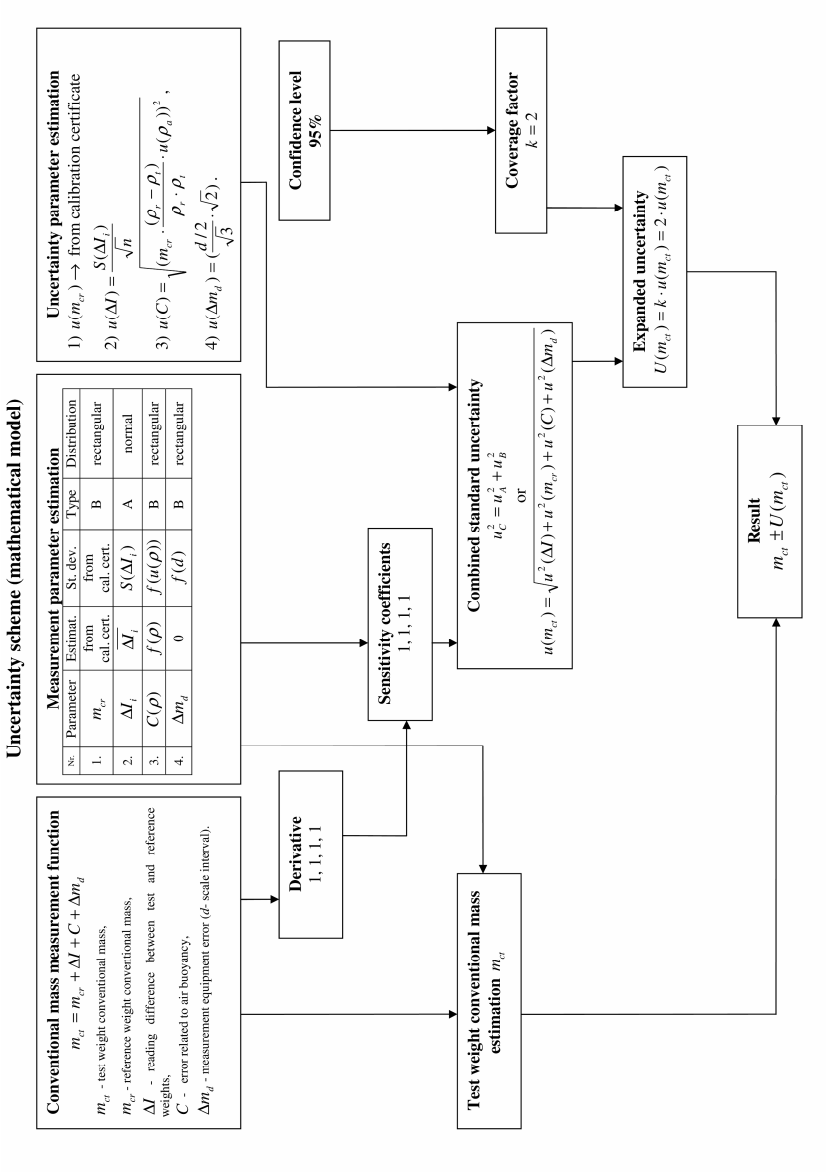


Fig. 5 Scheme of conventional mass measurement uncertainty

During high accuracy measurements not one measurement is made and not even two measurements. Instead, several cycles of measurements are made. Regular measurement cycle is “ABBA” where A is standard weight, and B is test weight or object. First cycle is “empty”, and its results are discarded. After that, several working cycles are performed. Measurement process (several cycles) can last quite long (half an hour or longer).

Results of ABBA cycles must be checked. For instance, results of any single cycle should not differ too much from each other and from previous comparator calibration results. Otherwise results of this measurement (results of several measurement cycles) are discarded. If measurement cycles results have passed examination, then they are averaged and several corrections are made (air density, comparator adjustment results etc.) These computations are made by computer that is connected to comparator.

Comparators are mainly used when highest accuracy of mass measurements is needed. This includes national and international metrology laboratories and some enterprises. Price of high accuracy comparators is very high – several tens thousands of euros. There are two main manufacturers of comparators - “Sartorius” (Germany) and “Mettler Toledo” (Switzerland).

Let’s compare metrological specifications of typical Sartorius comparator and Sartorius analytical scales. Sartorius CC310 mass comparator has measurement range till 200 g, discretion is 0.01 mg and standard deviation is 0.01 mg. It allows calibration of weights of E_1 (highest) accuracy class. Professional analytical scales Sartorius LA230S have measurement range till 230 g, discretion is 0.1 mg and standard deviation is 0.1 mg.

For more convenient process of measurement auxiliary computer software has been written for mass measurement. Using this software, mass comparator can be controlled by computer. Moreover, these programs read measurement data, perform calculations and analysis, and automatically prepare results protocol and calibration certificate. National Metrology centre of Latvia uses “Scales Net32” software that is made by German company “Häfner Gewichte GmbH, Maro-Elektronik”.

Chapter 3: WEIGHTING METHODS FOR INTERLABORATORY COMPARISON

Third chapter describes weighting methods. In past, following methods were used in laboratories to determine conditional mass of the weight with the help of non-automatic scales: double weighting method (Gauss method), weighting method with one shoulder (Borda method) and Mendeleev method (weighting with constant load on one shoulder).

Lots of attention was paid to weighting methods that laboratories use nowadays. There are three different accepted measurement cycle procedures

that are meant for one comparative measurement. For this, other procedures and measurement circles can be used. If measurement cycles are dependant on each other (for example, $A_1\ B_2\ A_2$; $A_2\ B_2\ A_3$) then measurement uncertainty is determined using covariance constraints. In the description of measurement cycle A denotes measuring reference weight and B denotes measuring test weight. Cycles ABBA and ABA usually are used to calibrate weights of E and F accuracy class. Circle $AB...B_nA$ often is used to calibrate weights of M accuracy class but it is not recommended for use with weights of E and F accuracy class. However, in case when comparator with automatic mechanism for weights shifting is used and measurement is made in protective camera then this cycle can also be used for calibrating weights of E and F accuracy class. To conduct set weighting method it is recommended to use ABBA and ABA measurement cycles. In case if several reference standards of mass are used for comparison then is its possible to conduct measurement cycle with every reference weight and then compare them to each other.

In order to compare two weights of E and F accuracy class it is recommended to use ABBA and ABA schemes because it allows compensation linear drift.

Calculation of conventional mass for weights of E and F accuracy class using cycle ABBA ($r_1\ t_1\ t_2\ r_2$):

$$I_{r1\_1}, I_{t1\_1}, I_{t2\_1}, I_{r2\_1}, \ldots\ldots\ldots, I_{r1\_n}, I_{t1\_n}, I_{t2\_n}, I_{r2\_n}$$

$$\Delta I_i = (I_{t1\_i} - I_{r1\_i} - I_{r2\_i} + I_{t2\_i})/2 \qquad (1)$$

where: $i=1, \ldots, n$

Calculation of conventional mass for weights of E and F accuracy class using cycle ABA ($r_1\ t_1\ r_2$):

$$I_{r1\_1}, I_{t1\_1}, I_{r2\_1}, \ldots., I_{r1\_n,}, I_{t1\_n}, I_{r2\_n}$$

$$\Delta I_i = I_{t1\_i} - (I_{r1\_i} + I_{r2\_i})/2 \qquad (2)$$

where: $i=1, \ldots, n.$

In the description of cycles ABBA and ABA schemes n denotes number of measurements. Denotations i are given according to the sequence of weights placed on the scales platform. Indexes r and t denote reference and test weights. Element ΔI_i denotes difference between values obtained during measurement sequence i.

Time intervals between measurements should be equal. If it is necessary to determine sensitivity of the scales used in the calibration process then scheme ABBA can be changed like this: I_r, I_t, I_{t+ms}, I_{r+ms}, where m_s is a weight that is used to determine sensitivity of the scales.

Comparison of several test weights of the same nominal mass with one standard weight can be done according to the scheme $AB_1...B_nA$ (it is recommended for M accuracy class scales).

If several test weights are calibrated at the same time $t(k)$ $(k=1...K)$ and these weights have equal nominal mass then measurement cycle ABA can be changed to $AB_1...B_nA$ like this:

Calculation of conventional mass for weights of M accuracy class using cycle $AB_1...B_nA$:

$$I_{r1\_1}, I_{t1\_1}, I_{t2\_1}, \ldots, I_{t(K-1)\_1}, I_{t(K)\_1}, I_{r2\_1},$$

$$I_{r1\_2}, I_{t1\_2}, I_{t2\_2}, \ldots, I_{t(K-1)\_2}, I_{t(K)\_2}, I_{r2\_2},$$

$$\ldots$$

$$I_{r1\_i-1}, I_{t1\_i-1}, I_{t2\_i-1}, \ldots, I_{t(K-1)\_i-1}, I_{t(K)\_i-1}, I_{r2\_i-1},$$

$$I_{r1\_i}, I_{t1\_i}, I_{t2\_i}, \ldots, I_{t(K-1)\_i}, I_{t(K)\_i}, I_{r2\_i}.$$

$$\Delta I_{i(k)} = I_{t(k)\_i} - (I_{r\_1} + I_{r\_(i+1)})/2 \qquad (3)$$

where $i=1,...,n$

If scales show insignificant drift (less than one third of required measurement uncertainty) then it is not necessary to change sequence of test weights in the scheme $AB_1...B_nA$ for repeated measurements. Number of test weights should not exceed 5. Number ff measurements n is determined by requirements of measurement uncertainty, repetition of weights and results compatibility.

Using these measurement cycles it is possible to determine conventional mass of the weight and to calculate measurement uncertainty.

Measurement uncertainty is the component of measurement results that denotes range of values that include true value. Measurement uncertainty includes all errors (both random and systematic errors) and therefore measurement uncertainty is the best way to express accuracy and precision of measurements. But sometimes systematic error is not taken into consideration and only occasional error is observed when calculating measurement uncertainty. If only occasional error is used in calculations then measurement precision is expressed.

See conventional mass measurement uncertainty calculation scheme on Fig. 5.